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Indian Standard

SPECIFICATION FOR PENTAERYTHRITOL

(First Revision)

UDC 547·427·1

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *July* 1988

Indian Standard

SPECIFICATION FOR PENTAERYTHRITOL

(First Revision)

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 17 December 1987, after the draft finalized by the Organic Chemicals (Misc) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was published in 1974. The Committee responsible for the preparation of this standard decided to revise it in view of the improved quality of the material which is currently being made available. In the present version the requirement of monopenta and dipenta content, and melting point have been updated. The test methods for monopenta content and colour of monoglyceride have been suitably modified.

0.3 Pentaerythritol is mainly used in the manufacture of surface paint coatings, printing inks and explosives industry. This specification does not cover nitration grade of the material which is used in explosives (*see* **1.2**). A separate specification for pentaerythritol for explosive industry, namely, IS : 10977-1984 has been prepared by Explosives and Pyrotechnics Sectional Committee.

0.4 In the preparation of this Standard, considerable assistance has been derived from ASTM D 2195-79. Standard Methods for Testing Pentaerythritol and ASTM D 2999-71 Standard Test Methods for Monopentaerythritol.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for pentaerythritol.

1.2 It does not cover nitration grade pentaerythritol used for explosive industry (*see* **0.2**).

2. REQUIREMENTS

2.1 The material shall be in the form of white free-flowing powder and free from foreign matter.

2.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of Table 1.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in polyethylene-lined jute or paper bags or drums as agreed to between the purchaser and the supplier.

TABLE 1 REQUIREMENTS FOR PENTAERYTHRITOL

(*Clause* 2.2)

Sl No. (1)	Characteristic (2)	Requirement (3)	Methods of Test (Ref to Cl No. in Appendix A) (4)
i)	Pentaerythritol (Monopenta) content, percent by mass (on dry basis), *Min*	92·0	A-3
ii)	Dipenta content inclusive of polypenta content, percent by mass (on dry basis), *Max*	8·0	A-3
iii)	Loss on drying, percent by mass, *Max*	0·50	A-4
iv)	Ash content, percent by mass, *Max*	0·10	A-5
v)	Melting point, *Min*	234·0°C	A-6
vi)	Hydroxyl value, percent by mass, *Min*	45·0	A-7
vii)	Colour of pentaerythritol phthalate	To pass the test	A-8
viii)	Colour of the monoglyceride with linseed oil	To pass the test	A-9
ix)	Sulphates	To pass the test	A-10
x)	Chlorides	To pass the test	A-11
xi)	Solubility	To pass the test	A-12

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and recognized trade-mark, if any;

c) Net mass of the material; and

d) Lot or batch number in code or otherwise.

3.2.1 The container may also be marked with the Standard Mark.

Note — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING AND CRITERIA FOR CONFORMITY

4.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR PENTAERYTHRITOL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in the test.

Note — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PREPARED SAMPLE

A-2.1 Weigh about 100 g of the material and dry for 30 minutes in an oven at a temperature of 110 ± 2°C. Cool in a desiccator and weigh. Dry again and repeat until constant mass is attained.

A-3. DETERMINATION OF PENTAERYTHRITOL (MONOPENTA CONTENT)

A-3.0 Two methods, namely, Method A and Method B, have been prescribed. Method A shall be used in case of dispute.

A-3.1 Method A (Benzaldehyde Method)

A-3.1.0 *Principle* — Pentaerythritol reacts with benzaldehyde in the presence of strong acid to form dibenzylacetal.

$$C(CH_2OH)_4 + 2\ C_6H_5CHO \rightarrow C_6H_5CH(OCH_2)_2C(OCH_2)_2CHC_6H_5$$

The dibenzylacetal so formed is filtered off, washed and dried to constant mass. A correction is made for the slight solubility of dibenzylacetal under the conditions of the test. Dipentaerythritol and polypentaerythritols which may be present in the sample do not react.

A-3.1.1 *Apparatus*

A-3.1.1.1 *Sintered glass crucible* — of porosity 3 and capacity 30 ml.

A-3.1.2 *Reagents*

A-3.1.2.1 *Benzaldehyde reagent* — Mix 1 volume of analytical reagent grade benzaldehyde with 5 volumes of anhydrous methanol.

A-3.1.2.2 *Methanol-water wash solution* — Mix 1 volume of methanol with 1 volume of water.

A-3.1.2.3 *Concentrated hydrochloric acid* — conforming to IS : 265-1976*.

A-3.1.3 *Procedure* — Accurately weigh about 0·5 g of the *prepared sample* (*see* **A-2.1**) into a 250 ml conical flask. Add 5 ml of water and heat to boiling to dissolve the sample. To the hot solution, add 20 ml of benzaldehyde reagent and mix the solution by swirling. Immediately add 12 ml of concentrated hydrochloric acid and mix again by swirling for 5 minutes. Stopper the flask and allow to stand at room temperature for 30 minutes. Cool the flask in an ice-bath for at least 1 hour with occasional swirling. Dilute the reaction mixture with 25 ml of ice-cold methanol-water wash solution. Filter the reaction mixture through a dried and weighed G3 sintered crucible, using a glass rod to complete the transfer. Wash the flask using 10 ml portions (total volume 100 ml) of ice-cold wash-solution to ensure complete transfer of the insoluble acetal-precipitate from the flask to the crucible. Dry the crucible with the precipitate to constant mass at 100 ± 2°C.

A-3.1.4 *Calculation*

Pentaerythritol (monopenta content), percent by mass

$$= \frac{(M_1 + 0{\cdot}026\,9) \times 43{\cdot}59}{M_2}$$

where

M_1 = mass in g of the precipitate, and

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*Specification for water for general laboratory use (*second revision*).

*Specification for hydrochloric acid (*second revision*).

M_2 = mass in g of the material taken for the test.

A-3.1.4.1 Dipenta content (inclusive of polypenta content), percent by mass = 100 − monopenta content.

NOTE 1 — 0·026 9 is the correction taken for calculation as the precipitate is soluble in methanol/water mixture at the cold temperature.

NOTE 2 — The benzaldehyde method of determination of purity of PE gives a slightly higher value as the PE formals also get precipitated along with the mono PE in the benzaldehyde complex. Hence, a correction has to be made for the PE formals by subtracting the value for PE formals obtained by the GLC method.

A-3.2 Method B (*p*-Nitrobenzaldehyde Method)

A-3.2.1 *Reagents*

A-3.2.1.1 p-*Nitrobenzaldehyde reagent*—Dissolve 1 g of *p*-nitrobenzaldehyde in 5 ml of methanol and 2 ml of concentrated hydrochloride acid. Heat to boiling under reflux on water-bath for 1½ hours.

A-3.2.1.2 *Methanol* — conforming to IS : 517-1967*.

A-3.2.1.3 *Concentrated hydrochloric acid* — conforming to IS : 265-1976†.

A-3.2.1.4 *Standard sodium hydroxide solution* — 1 N.

A-3.2.1.5 *Phenolphthalein indicator* — 0·5 percent (*m/v*). Dissolve 0·5 g of phenolphthalein in 100 ml of rectified spirit (*see* IS : 323-1959‡) and make it faintly pink by the addition of dilute sodium hydroxide solution.

A-3.2.2 *Procedure* — Weigh accurately 2 g of the *prepared sample* (*see* **A-2.1**) into a 100-ml conical flask, dissolve in minimum quantity of water and dilute to the mark. Mix 10 ml of this solution with 1 g of *p*-nitrobenzaldehyde reagent in a 250-ml round-bottom flask fitted with ground glass stopper. Cool and neutralize the contents to phenolphthalein with sodium hydroxide solution. Add sufficient quantity of methanol to make a 75 percent solution of the sample with methanol. Boil the contents for a short time, cool and filter under vacuum through previously weighed sintered glass crucible. Wash the residue with 60 ml of 75 percent methanol and dry to constant mass at 110°C. Cool in a desiccator and weigh.

A-3.2.3 *Calculation*

Pentaerythritol (monopenta content), percent by mass

$$= \frac{0{\cdot}338\ 37\ M}{M_1} \times 100$$

where

M = mass in g of the residue obtained, and

M_1 = mass in g of the material taken for the test.

A-4. DETERMINATION OF LOSS ON DRYING

A-4.0 Outline of the Method — A known mass of of the sample is taken in a tared aluminium dish and dried at 105°C for 3 hours. Loss in mass is obtained.

A-4.1 Apparatus

A-4.1.1 *Tared Aluminium Dish*

A-4.1.2 *Air-oven* — preferably electrically heated with temperature control device.

A-4.1.3 *Desiccator* — Containing an efficient desicant.

A-4.2 Procedure — Weigh accurately about 5 g of the sample into a tared aluminium dish and place in the oven maintained at 105 ± 2°C for 3 h. Remove the dish from the oven, cool in a desiccator to room temperature and weigh. Repeat this procedure. Keep the dish in the oven only for half an hour each time until the difference between two successive weighings is less than 1 mg.

A-4.3 Calculation

$$\text{Loss on drying, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M_1 = loss in mass in g of the sample upon drying, and

M = mass in g of the sample taken for the test.

A-5. DETERMINATION OF ASH

A-5.1 Apparatus

A-5.1.1 *Platinum or Silica Basin*

A-5.1.2 *Furnace* — capable of maintaining temperature within 800 + 25°C .

A-5.2 Procedure — Slowly burn, in several portions, approximately 50 g of the material, weighed to the nearest 1 g, in a tared platinum or silica basin and ignite finally in a furnace at 800 ± 25°C until all volatile matter has disappeared. Cool in a desiccator and weigh.

A-5.3 Calculation

$$\text{Ash, percent by mass} = \frac{M}{M_1} \times 100$$

where

M = mass in g of the residue after ignition, and

M_1 = mass in g of the material taken for the test.

A-6. DETERMINATION OF MELTING POINT

A-6.1 Procedure — Carry out the determination of melting point according to the method prescribed in IS : 5762-1970*.

A-7. DETERMINATION OF HYDROXYL VALUE

A-7.0 Two methods, namely, Method A and Method B have been prescribed. Method A shall be used in case of dispute.

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*Specification for methanol (methyl alcohol) (*first revision*).

†Specification for hydrochloric acid (*second revision*).

‡Specification for rectified spirit (*revised*).

*Methods for determination of melting point and melting range.

A-7.1 Method A

A-7.1.0 *Principle* — The sample is acetylated with acetic anhydride and sodium acetate followed by saponification of the resulting acetyle product. Since the acetylated polypentaerythritols are not readily soluble in water, a little acetone is added to effect solution.

A-7.1.1 *Apparatus*

A-7.1.1.1 *Round-bottom flasks* — 200-ml and 1 000-ml capacities fitted with ground glass joints and air condensers.

A-7.1.2 *Reagents*

A-7.1.2.1 *Sodium acetate, anhydrous* — analytical reagent grade.

A-7.1.2.2 *Acetic anhydride* — analytical reagent grade.

A-7.1.2.3 *Standard sodium hydroxide solution* — 1 N.

A-7.1.2.4 *Standard hydrochloric acid solution* — 1 N.

A-7.1.2.5 *Acetone* — analytical reagent grade.

A-7.1.2.6 *Sodium hydroxide solution* — 0·5 N.

A-7.1.3 *Procedure* — Weigh accurately about 1·4 g of the *prepared sample* into a dry 200-ml flask and add 3 g of anhydrous sodium acetate and 7·5 ml of acetic anhydride. Connect the flask to a vertical condenser and heat on sand-bath so that the liquid is kept boiling for 2 hours. Allow the flask to cool for about 15 minutes and add about 50 ml of freshly boiled water at 80°C to wash down the condenser. Shake the flask to dissolve as much of the product as possible retaining the temperature not above 80°C. Cool the flask and transfer the solution, without filtering, to a 1 000-ml round-bottom flask. Dissolve any product remaining in the acetylation flask in 10 ml of acetone added in several small portions from a cylinder, and transfer to the 1 000-ml flask. Add 2 ml of phenophthalein indicator and carefully neutralise the solution by the slow addition of sodium hydroxide solution and with constant stirring to avoid local saponification.

Finally, wash the side of the flask with boiled water and add 50 ml of standard sodium hydroxide solution. Attach the flask to a reflux condenser and boil gently for 1 hour. Stopper the flask, cool rapidly and titrate the excess of standard sodium hydroxide with standard hydrochloric acid.

A-7.1.3.1 Carry out a blank test omitting only the pentaerythritol and using 5 ml of standard sodium hydroxide solution for the saponification instead of 50 ml.

A-7.1.4 *Calculation*

Hydroxyl value, percent by mass

$$= \frac{(9\,V_3 - 10\,V_1 + 10\,V_2)\,0{\cdot}170}{M_1}$$

where

V_3 = volume in ml of standard hydrochloric acid equivalent to 50 ml of standard sodium hydroxide solution,

V_1 = volume in ml of standard hydrochloric acid required for titration with the sample solution,

V_2 = volume in ml of standard hydrochloric acid required for the blank, and

M_1 = mass in g of the material taken for the test.

A-7.2 Method B

A-7.2.1 *Reagents*

A-7.2.1.1 *Standard sodium hydroxide solution* — 0·5 N.

A-7.2.1.2 *Acetylation reagent* — Add 12 ml of acetic anhydride to 88 ml of pyridine containing 0·3 to 0·4 percent water content and mix well.

A-7.2.1.3 *Phenolphthalein indicator* — 0·5 percent (*m*/*v*). Dissolve 0·5 g of phenolphthalein in 100 ml of rectified spirit (*see* IS : 323-1959*) and make it faintly pink by the addition of dilute sodium hydroxide solution.

A-7.2.2 *Procedure* — Weigh accurately 0·5 g of the *prepared sample* in a 250-ml round-bottom flask fitted with ground stopper. Pipette into this exactly 20 ml of acetylation reagent. Attach reflux condenser and reflux the mixture on an oil-bath at a temperature of 120-130°C for ½ hour. Flush the condenser with water and rinse glass joint before removing the flask. Cool in an ice-bath and titrate against standard sodium hydroxide solution using phenolphthalein indicator. Also carry out a blank determination.

A-7.2.3 *Calculation*

Hydroxyl value, percent by mass

$$= \frac{1{\cdot}70\,(V_1 - V_2) \times N}{M}$$

where

V_1 = volume in ml of standard sodium hydroxide solution used in the blank,

V_2 = volume in ml of standard sodium hydroxide solution used in the titration,

N = normality of sodium hydroxide solution, and

M = mass in g of the sample taken for the test.

A-8. DETERMINATION OF COLOUR OF PENTAERYTHRITOL PHTHALATE

A-8.1 Apparatus

A-8.1.1 *Thermostatically Controlled Oil-Bath* — Provided with stirrer and a lid fitted with holes to take at least four 15 × 2·5 cm test-tubes.

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*Specification for rectified spirit (*revised*).

A-8.1.2 *Pyrex Glass Test Tubes* — 25 × 150 mm.

A-8.2 Reagents

A-8.2.1 *Phthalic Anhydride* — AR grade.

A-8.2.2 *Standard Colour Solution* — *see* IS : 8768-1978*.

A-8.3 Procedure — Weigh 1 g of previously dried pentaerythritol and mix thoroughly with 1 g of phthalic anhydride in a glass pestle and mortar. Place the mixture in pyrex glass test tube and heat to 200°C in thermostatically controlled oil bath. Maintain the temperature for seven minutes. Allow the mixture to cool to room temperature. Compare the colour of this mixture with standard colour solutions prepared separately (**A-8.2.2**) by visual observation against a white background.

A-8.3.1 Report the colour of the sample nearest to the colour standard in Hazen units. In the event of the colour lying between two values, the value nearer to the darker side is to be reported.

A-9. DETERMINATION OF COLOUR OF MONOGLYCERIDE

A-9.1 Reagent

A-9.1.1 *Linseed Oil* — conforming to IS : 75-1973†.

A-9.1.2 *Litharge or Hydrated Lime*

A-9.2 Procedure — Charge 150 g of oil in a 3-necked flask fitted with a stirrer and a thermometer. Heat the oil to 120°C and add litharge and raise the temperature to 250°C in inert gas and add 5 g of pentaerythritol. This will lower the temperature considerably. Raise temperature again to 250°C and add another 5 g of pentaerythritol. Raise temperature to 250°C and maintain at 250 ± 2°C for 45 minutes. Time should be noted from the time the mass reaches 250°C after the completion of addition of pentaerythritol. The quantity of pentaerythritol added should be such that the monoglyceride formed is in the mass ratio of pentaerythritol to linseed oil as 1 : 15. Cool the monoglyceride to room temperature. Carry out the same procedure under similar conditions using the same quantity of linseed oil and litharge without adding pentaerythritol. Cool it to room temperature and compare its colour with monoglyceride in Lovibond tintometer with 1 cm cell width or any other suitable instrument.

A-9.2.1 The material shall be taken to have passed the test if colour of monoglyceride is not darker by 10 units on Lovibond tintometer than that of linseed oil alone treated under similar conditions.

A-10. TEST FOR SULPHATES

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinder* — 50-ml capacity (*see* IS : 4161-1967‡).

A-10.2 Reagents

A-10.2.1 *Dilute Hydrochloric Acid* — approximately 5 N.

A-10.2.2 *Barium Chloride Solution* — Dissolve 12 g of barium chloride in water and make up to 100 ml.

A-10.3 Procedure — Dissolve 2·0 g of the material in 50 ml of water contained in the Nessler cylinder. Add 1 ml of dilute hydrochloric acid and 1 ml of barium chloride solution. Allow to stand for one hour.

A-10.3.1 The material shall be taken to have passed the test if no turbidity or precipitate is produced.

A-11. TEST FOR CHLORIDES

A-11.1 Apparatus

A-11.1.1 *Nessler Cylinder* — 50 ml (*see* IS : 4161-1967‡).

A-11.2 Reagents

A-11.2.1 *Dilute Nitric Acid* — approximately 5 N.

A-11.2.2 *Silver Nitrate Solution* — Dissolve 4·2 g of silver nitrate in 100 ml of water.

A-11.3 Procedure — Dissolve 2·0 of the material in 50 ml of water contained in the Nessler cylinder. Add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution.

A-11.3.1 The material shall be taken to have pased the test, if no opalescence is produced.

A-12. TEST FOR SOLUBILITY

A-12.1 Dissolve 1 g of the material in 12 ml of water. A clear colourless solution shall be obtained.

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*Method of measurement of colour in liquid chemical products in Hazen units.

†Specification for linseed oil, raw and refined (*second revision*).

‡Specification for Nessler cylinders.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF PENTAERYTHRITOL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable clean, dry and air-tight containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost but not completely filled by the sample.

B-1.7 Each sample container shall be sealed airtight after filling and marked with full identification particulars, such as sample number, the date of sampling, batch of manufacture of the material and other important particulars of the consignment.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not very unduly from the normal temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In a single consignment, all packages of the same size, containing material from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of packages belonging to different batches of manufacture, packages belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a lot.

B-2.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, samples shall be tested on each lot separately.

B-2.3 The number of packages to be sampled for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

B-2.3.1 These packages shall be selected at random. In order to ensure the randomness of selection, procedures given in IS : 4905-1968* may be followed.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the packages selected, draw with an appropriate sampling instrument small quantity of material from different portions of the package and mix thoroughly. This shall constitute the representative sample for the package.

TABLE 2 SCALE OF SAMPLING

(*Clause* B-2.3)

LOT SIZE N (1)	NUMBER OF CONTAINERS TO BE SELECTED n (2)
Up to 50	3
51 to 100	4
101 to 150	5
151 to 300	6
301 to 500	7
501 to 800	8
801 to 1 300	9
1 301 and above	10

B-3.2 From the samples representing different packages selected in small but approximately equal quantity of material shall be taken and mixed thoroughly to form composite sample which shall be sufficient for carrying out triplicate tests for the characteristics given in Table 1. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.3 The referee sample shall bear the seal of both the purchaser and the supplier and shall be kept at a place agreed to between the two. It shall be used in case of any dispute between the two.

B-3.4 The composite samples shall be transferred to separate containers. These sample containers shall then be sealed with stoppers and labelled with full identification particulars given in **B-1.7**.

B-4. NUMBER OF TESTS

B-4.1 Tests for all characteristics given in Table 1 shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to this specification if the test results on the composite sample for each of the characteristics meet relevant requirements given in Table 1.

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*Methods for random sampling.

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